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NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

by

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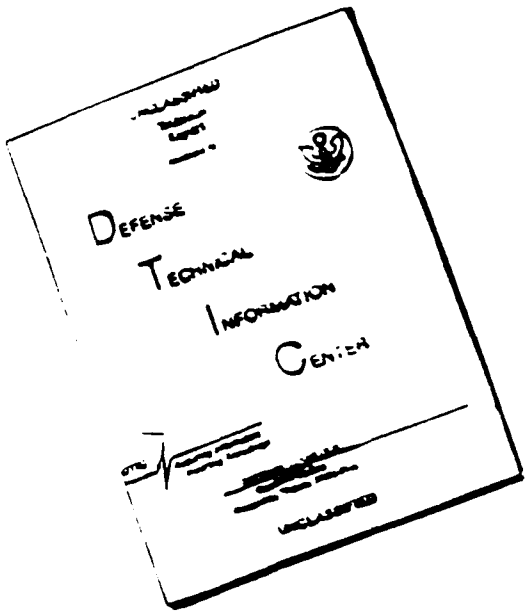

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Nuclear Magnetic Resonance Spectroscopy

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TXT03
SEN03 1
PAR03

INTRODUCTION AND SCOPE

SEN03 1 NMR continues to grow and find new applications. A
SEN06 3 maturing understanding of spin physics has allowed the de-
11 velopment of sophisticated techniques for assigning reso-
17 nances, determining internuclear distances, and averaging or
SEN09 24 exploiting orientation-dependent interactions. The recent and
5 continuing revolution in biotechnology would not be possible
13 without the increasingly elegant NMR methods that are used
22 routinely to characterize the structure and dynamics of pro-
30 teins and nucleic acids and their interactions with other
SEN12 39 biomolecules. NMR imaging has revolutionized diagnostic
7 medicine and is beginning to have an impact on nondestructive
SEN15 17 materials testing. Spatially localized spectroscopy is revealing
7 the chemistry of the life process noninvasively.

PAR06
SEN03 1 The few remaining obstacles to high-resolution NMR
SEN06 9 spectroscopy of solids are falling rapidly. Multidimensional
3 solid-state NMR experiments are revealing the structures of
SEN09 11 catalysts. High-temperature catalytic reactions are being
7 simulated in NMR probes so that reaction mechanisms may
SEN12 16 be elucidated. NMR studies of high-T_c superconductors have
8 been underway in a number of laboratories since shortly after
SEN15 18 the discovery of the prototypical materials. NMR spectros-
3 copy has proven itself to be exceptionally adaptable to new
SEN18 13 problem areas. A good example of this was recently provided
10 by Yannoni who accurately measured the bond distances in
19 C_{60} months before the first crystal structure of a fullerene
29 derivative was determined.

PAR09
SEN03 1 NMR spectroscopy continues to be invaluable as a routine
11 technique for the study of structure and dynamics of organic
SEN06 21 and inorganic compounds. A significant fraction of the articles
8 in the chemical literature mentions data obtained by NMR
17 spectroscopy, if only in passing; and most chemists readily
26 appreciate why Richard Ernst was awarded the Nobel Prize
35 in chemistry for his contributions to NMR spectroscopy.

PAR12
SEN03 1 My predecessor surveyed the literature through March of
SEN06 10 1990. A search of *Chemical Abstracts* revealed 6954 publi-
9 cations on NMR spectroscopy between April 1990 and De-
17 cember 1991 and 7936 publications for the entire biennium.
SEN09 1 The corresponding totals for 1970 plus 1971 and 1980 plus
SEN12 12 1981 were 3186 and 5808, respectively. NMR spectroscopy
SEN15 4 as a scholarly enterprise is clearly in good health. Ironically,
3 the number of NMR articles published in this journal is down
SEN18 14 from 29 in 1980–1981 to 18 in 1990–1991. It is clearly im-
5 possible to cite all 6954 publications, and to do so nonselec-
SEN21 15 tively would probably not be much of a contribution. I have
SEN24 4 emphasized a chemist's perspective. I have completely ignored
6 clinical work and largely ignored in vivo experiments and
SEN27 15 studies of excised tissue. I have tried to give a balanced
9 coverage of the quickly expanding literature on proteins and
SEN30 18 nucleic acids in solutions. The coverage of imaging and
7 spatially localized spectroscopy emphasizes technique de-
12 velopment and materials science applications rather than
SEN33 19 human or animal studies. The coverage of NMR spectroscopy
7 of solids and chemical applications thereof is probably the
16 most complete, reflecting my better knowledge of that liter-
24 ature.

PAR15
SEN03 1 The division of topics is necessarily arbitrary. For example,
SEN06 4 some of the citations in the Multidimensional NMR Spec-
12 troscopy section could have been placed in the Biomolecules
SEN09 21 in Solution section and vice versa. Even the division between
6 topics like Synthetic Polymers in Solution and Solid Polymers
15 seems arbitrary, but division is necessary even if only to reduce
26 the task of writing this review to a series of manageable steps.

TXT06

BOOKS

SEN03 1
PAR18
SEN03 1 A number of books on NMR spectroscopy have either been
12 published or caught the eye of other reviewers in the past 20
SEN06 24 months. In many cases, it was easier to find a book review
13 in the *Journal of the American Chemical Society*, *Journal*
22 *of Magnetic Resonance*, or some other publication than to get
SEN09 32 access to the book itself. Making a virtue of necessity, I have
SEN12 9 cited both the books and their respective reviews. There were
SEN15 4 quite a few additions to numbered series (*A1–A10*). Marshall
SEN18 3 published a new book on the Fourier transform (*A11*). Slichter
3 published a third edition of *Principles of Magnetic Resonance*

TXT06
PAR18
(*A12*), and several other texts were published and reviewed (*A13–A17*). Two books discussed biological applications (*A18*, *A19*). Others dealt with imaging (*A20*), polymer microstructure (*A21*), and ^{31}P (*A22*). Farrar published a second edition of his text *Introduction to Pulse NMR Spectroscopy* (*A23*) and the first volume of this treatment on density matrices (*A24*).

TXT09

CONCEPTS IN MAGNETIC RESONANCE

PAR21
A positive development in the NMR literature was the creation of the journal *Concepts in Magnetic Resonance* which is subtitled "An Education Quarterly". Each issue publishes three or four tutorials on various aspects of NMR spectroscopy. The level is variable, but is generally well suited to a second-year student specializing in NMR spectroscopy.

PAR24
Some of the articles which caught my eye included treatments of density matrices (*B1*, *B2*), instrumentation (*B3–B6*), CRAMPS (*B7*), shaped pulses (*B8*), imaging (*B9*), and chemical shift anisotropy (*B10*).

TXT12

INSTRUMENTATION

PAR27
The best way to learn of new instrumental developments in NMR spectroscopy is to attend the annual Experimental NMR Conference (ENC) and tour the poster sessions and vendor suites. The progress in instrumentation development in this field is understated by the relatively few papers that have a primarily instrumental focus. This is due in part to the tendency of spectrometer and probe vendors not to publish in the open literature. NMR spectroscopists also have a tendency to downplay the role of instrument development, relegating its description to a subordinate role in papers devoted to chemical or spectroscopic results or to presentations at the ENC.

PAR30
High pressures are sometimes desirable for forcing equilibrium or characterizing a variety of physical phenomena. Two improvements in sample tubes for high-pressure solution NMR spectroscopy have been described (*C1*, *C2*).

PAR33
One of the most important recent advances in solid-state NMR spectroscopy was the development of double rotation (DOR) and dynamic-angle spinning (DAS) for high-resolution studies of noninteger quadrupole nuclei. These techniques are extremely demanding on the design of the spinning systems. Pines and co-workers have described designs of DOR (*C3*) and DAS (*C4*) probes and their applications to selected problems (see sections on solids). Another spinning system suitable for DAS studies has also been described (*C5*). Yannoni and co-workers described the design of a magic-angle-spinning (MAS) probe that works at temperatures down to 5 K and used it to study carbocations at near-liquid-helium temperatures (*C6*, *C7*). Stebbins has reviewed high-temperature solid-state NMR spectroscopy (*C8*). Temperatures over 1000 K have been achieved with a laser heating system (*C9*), and more conventional high-temperature probe designs have also been reported (*C10*, *C11*). Inductive heating has also been proposed as a method for achieving very high temperatures (*C12*). An apparatus for achieving temperature jumps in a short time has also been reported (*C13*).

PAR36
Accurate measurement of sample temperatures in NMR spectroscopy can be a vexing problem. Refinements in ratio pyrometry have been reported that seem applicable to very high temperatures (*C14*, *C15*). Chemical shift thermometry for MAS NMR spectroscopy and its application to measuring temperature gradients have received attention (*C16–C18*). Low-temperature probe designs for dilution refrigerator (*C19*) and matrix isolation experiments were also described (*C20*).

PAR39
A spinning speed controller for MAS experiments was described (*C21*) as were several useful radio-frequency devices (*C22–C24*). A spectrometer based on a dc SQUID that is suitable for NQR and low-frequency NMR spectroscopy has been developed (*C25*).

TXT15

DATA PROCESSING, CALCULATIONS, AND SIMULATIONS

PAR42
The data processing demands of modern NMR experiments continue to grow as a result of multidimensional spectroscopy, computationally-intensive alternatives to Fourier transformation, distance-geometry calculations from NOE data, etc. This has been reflected in two trends in data processing hardware. Spectrometer vendors are starting to move away from in-house data systems to generic interfaces that connect to commercial high-performance workstations. Secondly, data processing is increasingly performed off-line. Indeed, several

TXT15
PAR42
4 fairly sophisticated NMR data processing packages are
SEN18 11 available that run on PCs and McIntosh computers. As NMR
4 spectroscopists begin to make use of recent advances in com-
13 putational chemistry, techniques such as molecular mechanics
20 and ab initio calculations will be integrated with experimental
SEN21 29 results. This section reviews recent developments in data
9 processing and other computer-intensive reports including
15 spectral simulation, interpretation, and chemical shift calcu-
21 lations.

PAR45
SEN03 1 NMR data processing was reviewed in detail by Hoffman
SEN06 11 and Levy (*D1*). Several parts of an introductory review on
9 the Fourier transform have appeared in *Journal of Chemical*
SEN09 18 *Education* (*D2–D4*). Improvements in multidimensional FT
SEN12 6 data processing have also been discussed (*D5*, *D6*). Both linear
4 prediction and maximum entropy methods have been explored
12 as alternatives to Fourier transformation, and these methods
SEN15 20 have been reviewed (*D7*). Many workers regard the im-
6 provements in resolution and sensitivity provided by maxi-
SEN18 13 mum entropy to be purely cosmetic. Jones and Hore have
SEN21 6 critiqued this method (*D8*, *D9*). Several improvements in
5 linear prediction methods have been reported (*D10*, *D11*).
SEN24 1 Even standard nonlinear least squares has been considered
SEN27 10 as an alternative to Fourier transformation (*D12*). If the FT
5 is used (as it still is in almost all cases), the resulting spectrum
18 requires phase correction; and new algorithms for doing this
27 automatically have been reported (*D13*, *D14*).

PAR48
SEN03 1 Computer methods are being used for structural elucidation
10 (*D15*) including the application of neural networks (*D16*) and
SEN06 19 graph theory (*D17*, *D18*). Chemical shifts have been analyzed
7 or predicted using structure–property relationships or other
SEN09 14 methods (*D19–D30*). First-principles calculation of chemical
6 shifts have been carried out using ab initio or other molecular
17 orbital methods (*D31–D38*).

PAR51
SEN03 1 Several unrelated computer-intensive studies that also
SEN06 8 deserve citation include the following. A statistical method
5 for correcting for finite spinning speeds in magic-angle-spin-
SEN09 12 *ning spectra* was reported (*D39*). A procedure for interactive
SEN12 6 product–operator calculations was described (*D40*). Ernst has
4 discussed computer-optimized TOSCY experiments (*D41*).
SEN15 1 Finally, a method to evaluate the octane number of gasolines
12 from ^{1}H spectra was reported (*D42*).

TXT18
SEN03 1 ## RELAXATION PHENOMENA

PAR54
SEN03 1 Relaxation is at least an incidental issue in many of the
SEN06 13 papers cited in other sections of this review. Several stand-out
SEN09 4 papers dealing mainly with relaxation are cited here. Anet
3 and co-workers have shown that there can be an antisymmetric
13 component of the shielding tensor that can, under special
22 circumstances, result in the surprising observation of $T_2 > T_1$
SEN12 32 (*E1*). Interesting relaxation effects in solids were reported
SEN15 9 (*E2–E5*). Farrar has been exploring differential line broad-
8 ening and other relaxation phenomena in solution (*E6–E8*),
16 and related ideas have been applied to macromolecules (*E9*)
25 and membrane structure (*E10*).

PAR57
SEN03 1 Several unrelated relaxation studies also caught my eye.
SEN06 1 Relaxation rate matrix analysis was discussed for interproton
10 distance determination (*E11*), T_2 processes in porous rocks
18 were studied (*E12*), T_1 was used to probe surface viscosity
28 (*E13*), and field-cycling relaxation spectroscopy was used to
SEN09 36 study protein backbone fluctuations (*E14*). Other contribu-
3 tions relating to relaxation theory included a treatment of
12 relaxation under continuous rf fields (*E15*) and solutions of
21 the Bloch equations in the linear response approximation
29 (*E16*).

TXT21
SEN03 1 ## MISCELLANEOUS DEVELOPMENTS IN SOLUTION NMR SPECTROSCOPY
5

PAR60
SEN03 1 NMR pulse sequences were once composed nearly exclu-
SEN06 9 sively of rectangular pulses. Advances in rf electronics and
7 theoretical methods for obtaining shaped pulses tailored for
15 specific purposes have led to a continuum of designer pulse
SEN09 25 sequences. Shaped pulses can be designed using a variety of
11 methods including average Hamiltonian theory and iterative
SEN12 18 numerical simulations. Gezelter and Freeman have recently
7 reported the use of neural networks simulated on a serial
SEN15 17 computer for the design of shaped pulses (*F1*). The early
SEN18 4 results are very encouraging. Shaped pulses are commonly
6 used for selective excitation (*F2*, *F3*).

PAR63
SEN03 1 Molecular hydrogen exists in two isomers with a total spin
SEN06 12 of 1 (ortho hydrogen) or 0 (para hydrogen). In 1987 Wei-
4 tekamp proposed and later demonstrated that very large
12 enhancements in ^{1}H signal intensities could be obtained by
21 synthetically incorporating hydrogen enriched in the para

TXT21
PAR63
28 isomer into a product of interest immediately prior to the rf
SEN09 39 pulse. This effect is analogous to CIDNP and was given the
12 name PASADENA (para hydrogen and synthesis allows dy-
SEN12 19 namically enhanced nuclear alignment). Eisenbert has re-
4 viewed his own work with para-hydrogen-induced polarization
11 (*F4*), and Bargon and co-workers have demonstrated an
19 analogous effect with enriched ortho hydrogen (*F5*).
PAR66
SEN03 1 Another new twist that is in its infancy is a laser version
SEN06 14 of NMR spectroscopy on optical-phase conjugation. The
3 theory of this radically new form of spectroscopy has been
13 outlined by Evans (*F6*), and Warren has undertaken exper-
SEN09 21 imental verification. Ultrasound might offer a new way to
SEN12 9 manipulate relaxation times (*F7*). Very strong magnetic field
6 gradient pulses are being incorporated in a number of ex-
15 periments (*F8*), and high-resolution spectroscopy in a static
23 magnetic field gradient has been demonstrated (*F9*, *F10*).
SEN15 1 Improvements is cross polarization in solution have been
SEN18 10 reported (*F11–F13*). Bodenhausen has described a graphical
7 approach for understanding NMR experiments that is more
SEN21 15 intuitive than density–matrix methods (*F14*). Solvent sup-
SEN24 3 pression continues to be an important problem. This area has
5 recently been reviewed (*F15*).
TXT24
SEN03 1 ## MULTIDIMENSIONAL NMR SPECTROSCOPY
PAR69
SEN03 1 Multidimensional NMR spectroscopy used to be a fancy
SEN06 10 way of saying 2-D NMR spectroscopy. In the last several years
SEN09 7 it has come to include 3- and 4-D NMR spectroscopy. Just
3 as a 2-D experiment involves an evolution period and a de-
13 tection period, a 3-D experiment involves two evolution periods
SEN12 22 prior to detection. The three time periods correspond (after
SEN15 8 Fourier transformation) to the three frequency axes. The
3 development of higher-dimensional experiments is largely
9 motivated by the application of NMR spectroscopy to in-
17 creasingly more complicated problems in biological chemistry
SEN18 24 involving molecules of ever higher molecular weight. The
3 improved resolution afforded by multidimensional filtering
9 has allowed NMR structures to be determined for proteins
SEN21 18 of 150 residues or greater. Applications to protein structure
6 determination have been covered in two recent reviews (*G1*,
SEN24 15 *G2*). The use of 2-D NMR spectroscopy to study chemical-
SEN27 10 exchange kinetics has also been reviewed (*G3*). There were
4 a number of reports of applying 3-D (*G4–G11*) and 4-D
14 (*G12–G15*) NMR spectroscopy to proteins as well as appli-
22 cations of multidimensional NMR spectroscopy to nucleic
SEN30 29 acids (*G16–G19*). 2-D NMR spectroscopy was applied to
8 paramagnetic molecules (*G20*) and at high pressure to study
SEN33 17 phospholipid vesicles (*G21*). An improved method for 2-D
7 data collection was also proposed (*G22*).
TXT27
SEN03 1 ## BIOMOLECULES IN SOLUTION
PAR72
SEN03 1 There was a large number of applications of NMR to
SEN06 12 proteins, nucleic acids, and other biomolecules in solution. In
3 a review article such as this, one cannot hope to do justice to
SEN09 16 this large and diverse field. I have attempted to cite repre-
7 sentative work to highlight trends rather than provide a
16 comprehensive listing of citations.
PAR75
SEN03 1 NMR studies of proteins in solution were reviewed (*H1–*
SEN06 10 *H3*). The combination of NMR spectroscopy and molecular
9 mechanics or molecular dynamics has become common (*H4–*
SEN09 16 *H8*). NMR spectroscopy has been used to study protein
SEN12 10 folding (*H9–H12*), and metal binding (*H13–H17*). Studies of
4 binding to cytochrome *c* (*H18*, *H19*) and nucleic acids (*H20–*
SEN15 13 *H22*) have also been reported. Paramagnetic effects continue
SEN18 5 to be exploited in protein studies (*H23–H26*). Triple-reso-
2 nance studies have probed protein structure (*H27*, *H28*).
SEN21 1 Loops in nucleic acids were studied (*H29*, *H30*). Carbohy-
SEN24 2 drates were the subject of other investigations (*H31*, *H32*).
TXT30
SEN03 1 ## SYNTHETIC POLYMERS IN SOLUTION
PAR78
SEN03 1 This field has reached a high level of maturity. ^{13}C NMR
SEN09 4 analysis of polymers was recently reviewed (*I1*). ^{19}F analysis
SEN12 4 has been applied to Nafion (*I2*). The chemistry of poly-
5 phosphazenes and related materials has been studied (*I3–I5*).
SEN15 1 Many studies have dealt with microstructures (*I6–I10*) or
SEN18 10 conformation (*I11*, *I12*). 2-D NMR spectroscopy was used to
SEN21 8 probe polymer–polymer interactions (*I13*). A ^{13}C study of a
7 soluble polydiacetylene was reported (*I14*).
TXT33
SEN03 1 ## INORGANIC COMPOUNDS IN SOLUTION
PAR81
SEN03 1 One of the more interesting applications of NMR spec-
10 troscopy in recent years was the proposal by Hamilton and
20 Crabtree that ^1H T_1 measurements could be used to distin-
29 guish between classical and nonclassical transition-metal
SEN06 35 hydrides. Halpern and co-workers recently published a de-

TXT33
PAR81
tailed assessment of this method (*J1*) which also serves as a
SEN09 good overview of the relevant literature. Olah has published
SEN12 NMR studies of inorganic ions (*J2, J3*). Bell has studied
aluminosilicate and borosilicate solutions (*J4*), while Oster-
young has characterized molten salts (*J5*).
PAR84
SEN03 Variable-temperature and/or variable-pressure measure-
SEN06 ments have been used to characterize dynamics (*J6–J10*). ^{17}O
analysis has been applied to polyoxo complexes (*J11, J12*).
SEN09 ^{195}Pt NMR spectroscopy has been used to study the chemistry
SEN12 of anticancer agents (*J13–J16*). NMR analysis of various other
metal nuclei has been applied to a range of chemical problems;
a representative sampling is cited (*J17–J26*).
TXT36
SEN03

SOLIDS IN GENERAL

PAR87
SEN03 Some recent developments in NMR analysis of solids have
SEN06 been reviewed by Chmelka and Pines (*K1*). One of the more
interesting new experiments has been Tyko's development
SEN09 of zero-field NMR spectroscopy in high field (*K2, K3*). Several
approaches for the study of dynamics by multidimensional
spectroscopy or line shape analysis have been reported (*K4–*
SEN12 *K7*). An anomalous effect of MAS speed on chemical shifts
of cuprous halides has been interpreted as due to the Lorentz
SEN15 force (*K8*). Thankfully, this effect is restricted to ionic con-
ductors.
PAR90
SEN03 **Proton and Fluorine Spectroscopy.** The spectroscopy
SEN06 of abundant spins in solids (usually ^{1}H or ^{19}F) is often dom-
SEN09 inated by strong homonuclear dipolar couplings. Progress
continues to be made in coherent averaging by multiple pulse
SEN12 sequences and/or high speed MAS. Some of the most im-
portant applications of ^{1}H NMR analysis to solids are in the
SEN15 area of catalysis. Protons are typically chemically dilute on
oxide surfaces, and MAS at modest speeds usually suffices
SEN18 for high resolution. Thus, ^{1}H studies of solid catalysts are
reviewed in the Adsorption Phenomena and Catalysis section.
PAR93
SEN03 New developments in the spectroscopy of abundant species
in solids include pulse sequences for homonuclear dipolar
decoupling (*K9, K10*), elimination of probe ringing in mul-
tiple-pulse spectroscopy (*K11*), and a treatment of residual
SEN06 line widths under MAS (*K12, K13*). Applications of ^{19}F NMR
SEN09 spectroscopy of solids have been reviewed (*K14*). An im-
provement in ^{19}F CRAMPS has been described (*K15*), and
fast MAS ^{19}F analysis has been applied to materials related
SEN12 to dental enamel (*K16*). Chemical applications of ^{1}H
CRAMPS have also been published (*K17, K18*).
PAR96
SEN03 **Distance Measurements.** There has been a renaissance
SEN06 in the use of dipolar couplings to determine internuclear
SEN09 distances. These couplings are inversely related to the third
SEN12 power of the separation. Although nonspinning techniques
continue to be useful (*K19*), there have been several break-
throughs that have allowed information about dipolar cou-
plings to be preserved in high-resolution spectra obtained with
SEN15 MAS. Tyko has described the DRAMA sequence for "dipolar
SEN18 recovery at the magic-angle" (*K20*). Schaefer and co-workers
have developed a rapidly growing family of rotational-echo,
double-resonance (REDOR) experiments for measuring het-
SEN21 eronuclear dipolar couplings (*K21–K27*). Griffin and co-
workers have exploited the rotational resonance condition to
measure homonuclear couplings between spin pairs (*K28*).
SEN24 Cross polarization between isolated spin pairs has also been
proposed for heteronuclear distance determinations (*K29*).
SEN27 Several applications of the above techniques to distance de-
termination in biomolecules and C_{60} are cited in the sections
which follow.
PAR99
SEN03 **Spectral Assignment.** The development of techniques
SEN06 for spectral editing and heteronuclear correlation is proceeding
SEN09 vigorously. Burum has described the WIMSE technique for
distinguishing among CH, CH_2, and CH_3 groups in ^{13}C CP/
SEN12 MAS spectroscopy (*K30*). Sethi has described spectral editing
in a one-dimensional separated local field approach (*K31*).
SEN15 Two- (*K32*) and three-dimensional (*K33*) heteronuclear cor-
SEN18 relation experiments in solids have been performed. Dou-
ble-quantum filtering has been used to select resonances from
SEN21 pairs of dipolar-coupled spins (*K34*). An alternative approach
to spectral editing via "cross depolarization" has also been
proposed (*K35, K36*).
PAR102
SEN03 **Polarization.** Several papers have dealt with the polari-
SEN09 zation of the nuclear spins in solids. Very high polarizations
have been achieved from a photoexcited triplet state and
dynamic nuclear polarization (*K37*) or alternatively by optical
SEN12 pumping (*K38*). The Overhauser effect has been exploited
SEN18 in MAS spectra (*K39*). Hartmann-Hahn cross polarization
has been reexamined (*K40–K42*).
PAR105

TXT36
PAR105
SEN03 1 **Chemical Shift Anisotropy.** There is a lot of interest in
SEN06 9 further developing methods for recovering CSA data from high
SEN09 18 resolution MAS spectra. A variety of strategies for doing this
9 were proposed or refined during the period of the review
SEN12 19 (*K43–K50*). Chemical shift tensors were measured using
8 single-crystal methods (*K51*) and in the presence of dipolar
SEN15 17 couplings (*K52*). Refinements in the TOSS experiments for
8 sideband suppression were also reported (*K53–K55*).
PAR108
SEN03 1 **Coupling and Decoupling.** Scalar coupling is usually less
SEN06 7 important in solid-state vs solution-state NMR as a result of
17 the lower resolution, and in the case of scalar coupling to ^{1}H,
SEN09 29 the need to remove dipolar couplings. *J* couplings can be
6 important in ^{31}P MAS spectra and for many inorganic com-
SEN12 15 pounds. In the case of highly mobile systems such as weak-
11 ly-adsorbed molecules on catalysts, plastic crystals, inclusion
18 complexes, or elastomers, the NMR properties may be in-
26 termediate between solidlike and solution-like, and even *J*
SEN15 34 couplings to ^{1}H may be observed. Several articles focused on
SEN18 6 *J* coupling during the period of the review (*K56–K62*). Im-
2 proved strategies for ^{1}H dipolar decoupling in ^{13}C MAS NMR
12 were also proposed (*K63–K65*).
PAR111
SEN03 1 **Quadrupoles.** The development of DOR and DAS was
SEN09 9 described above. Several applications of DOR are cited in the
SEN12 10 section on Adsorption Phenomena and Catalysis. Pines has
SEN15 4 described pure adsorption phase DAS (*K66*). Most experi-
3 ments on quadrupoles used MAS or variable-angle spinning
SEN18 11 (VAS) (*K67–K75*). Several developments in ^{2}H spectroscopy
SEN21 7 were reported (*K76–K79*). The effects of dipolar coupling to
8 $I = 1$ nuclei on the spectrum of spin-$^{1}/_{2}$ nuclei were discussed
SEN24 20 (*K80–K82*). NQR with a dc SQUID was developed (*K83*).
TXT39
SEN03 1 ## ADSORPTION PHENOMENA AND CATALYSIS
PAR114
SEN03 1 The application of NMR methods to the study of catalysts
SEN06 12 and adsorbed species continues to grow. At first glance, NMR
6 spectroscopy might seem inappropriate to the study of surfaces
15 due to its low inherent sensitivity relative to traditional forms
SEN09 25 of surface spectroscopy. Indeed, NMR studies of two-di-
6 mensional surfaces have been few in number, and there has
16 been little effort to connect NMR studies to the single-crys-
SEN12 25 tal-face world of UHV surface science. The virtue of NMR
6 spectroscopy is that it is frequently applicable to the actual
SEN15 16 working catalyst with remarkable sensitivity. Amorphous
3 oxide and zeolite catalysts typically have surface areas mea-
SEN18 11 sured in tens or hundreds of square meters per gram. Since
3 radio waves readily penetrate through such catalysts, the
11 NMR experiment integrates over a considerable surface area.
SEN21 1 NMR spectroscopy is now being used in an in situ mode to
14 investigate the reaction mechanisms of various catalytic
21 processes.
PAR117
SEN03 1 NMR analysis has also made remarkable contributions to
10 the understanding of framework structures of zeolites and
SEN06 18 aluminum phosphate molecular sieves. Although most such
5 studies involve MAS, DOR was quickly applied to sharpen
14 the resonances of quadrupolar nuclei in catalysts.
PAR120
SEN03 1 This section also reviews NMR studies of surface-bound
10 alkylsilanes as well as clathrates and inclusion complexes.
PAR123
SEN03 1 **Reviews.** Slichter and co-workers have reviewed NMR
SEN06 8 techniques for the study of supported transition-metal cata-
SEN09 15 lysts (*L1*). Fyfe and co-workers reviewed the use of NMR
10 spectroscopy to determine zeolite lattice structure (*L2*).
SEN12 1 Klinowski reviewed molecular sieve catalysts (*L3, L4*). ^{1}H
SEN15 3 MAS studies were reviewed by Mastikhin and co-workers (*L5*).
PAR126
SEN03 1 **^{1}H Studies.** Magic-angle spinning alone generally suffices
SEN06 7 for reasonable resolution in studies of catalysts, and the re-
SEN09 16 sidual line width may simply reflect heterogeneity (*L6*). Other
3 ^{1}H studies dealt with acid sites and acidity (*L7–L11*), silanol
SEN12 13 groups (*L12*), and adsorbates (*L13, L14*). In a similar vein,
6 ^{19}F MAS NMR spectroscopy has also been applied to catalysts
16 (*L15*).
PAR129
SEN03 1 **Zeolite Frameworks.** The application of standard ^{29}Si
SEN06 7 and/or ^{27}Al MAS NMR spectroscopy to the study of frame-
SEN09 16 works is a routine characterization method (*L16–L24*). The
3 combination of NMR spectroscopy and X-ray powder dif-
SEN12 10 fraction can be fruitful (*L25–L27*). Fyfe and co-workers have
6 been determining bonding connectivities in zeolites by 2-D
SEN15 14 solid-state experiments (*L28–L33*). Gallium is sometimes
5 substituted for aluminum in zeolite synthesis, and $^{69/71}Ga$ MAS
14 NMR spectroscopy has been explored in their study (*L34,*
23 *L35*).
PAR132
SEN03 1 **Organic Adsorbates.** ^{13}C MAS NMR spectroscopy has
SEN06 7 been used to characterize reaction products on catalysts,

TXT39
PAR132
SEN09 15 3 SEN12 10 3 9 SEN15 15 8 SEN18 17 4

sometimes after progressive off-line heating (*L36–L41*). Haw and co-workers have studied catalytic reaction mechanisms with an in situ variable-temperature approach. Processes studied include reactions of unsaturated hydrocarbons (*L42–L44*), cracking reactions (*L45*), and methanol-to-gasoline chemistry (*L46, L47*). MAS NMR spectroscopy has also been used to study templates (*L48–L52*) and the effects of adsorbates on ^{29}Si spectra of zeolites (*L53*). Several studies addressed coke formation on catalysts (*L54–L58*).

PAR135
SEN03 1 SEN06 5 15 SEN09 24 SEN12 7 3 SEN15 13 4 12

Inorganic Adsorbates. Inorganic clusters and organometallic clusters on catalyst supports are of interest for their roles as catalysts, catalyst precursors, or other materials such as semiconductors. NMR spectroscopy was used to characterize a number of such materials (*L59–L65*). Molybdenum and vanadium oxides on alumina and silica were studied by ^{95}Mo and ^{51}V MAS NMR spectroscopy (*L66–L71*). ^{31}P MAS was used to characterize zeolite HZSM-5 modified with orthophosphoric acid (*L72*).

PAR138
SEN03 1 SEN06 7 SEN09 16 3 SEN12 12 SEN15 12 5 SEN18 11 SEN21 8 3 SEN24 12 7

Metal Surfaces. The NMR observation of species adsorbed on metal surfaces can be complicated by susceptibility problems, Knight shifts, and site heterogeneity. Several studies addressed CO adsorption on colloidal Pt or Pd (*L73–L75*). The observation of CO on a Pt electrode by NMR spectroscopy has also been reported (*L76*). CO and other adsorbates have been characterized on supported-metal catalysts (*L77–L80*). CO and other adsorbates have been characterized on supported-metal catalysts (*L77–L80*). NMR analysis of adsorbed hydrogen has been used to probe chemisorption (*L81–L86*). Promotor effects on supported metals have also been studied (*L87, L88*).

PAR141
SEN03 1 SEN06 4 12 SEN09 23 9 SEN12 18 10 SEN15 1 11 20

Aluminophosphate Molecular Sieves. The growing family of $AlPO_4$ frameworks and their derivatives prepared by substituting other elements for Al are well suited for NMR study. Pines' double rotation (DOR) technique has been applied to obtain high-resolution ^{27}Al spectra of these materials (*L89, L90*). ^{31}P and ^{27}Al MAS NMR spectroscopy has also been applied to characterize $AlPO_4$ frameworks (*L91–L96*). Adsorption of water and other species has been studied (*L97–L99*), and ^{129}Xe NMR analysis has been applied (*L100, L101*).

PAR144
SEN03 1 SEN09 8 4 13 SEN12 23 5 12 SE...3 19 SEN18 8 11 SEN21 18 SEN24 10 6 14

Other Applications. Cations in zeolites have been studied by ^{133}Cs, ^{23}Na, and other techniques (*L102–L106*). ^{129}Xe NMR spectroscopy has been applied as a probe by Dybowski, Fraissard, and others to address a number of problems in adsorption phenomena (*L107–L126*). In conjunction with multiple-quantum NMR spectroscopy, ^{129}Xe analysis has been applied to determine adsorbate heterogeneity in zeolite samples (*L127*). Amorphous carbons have been studied (*L128, L129*). Diffusion in catalysts has been studied by a pulsed field gradient (*L130, L131*) and tracer-exchange spectroscopy (*L132*). Acidity has been investigated by NMR analysis of probe molecules (*L133–L139*). The structure and dynamics of alkylsilyl-modified silica have been studied by various methods (*L140–L143*).

PAR147
SEN03 1 SEN06 4 SEN09 13 6

Clathrates and Inclusion Compounds. NMR studies of a variety of organic (*L144–L153*) and inorganic (*L154–L158*) guest molecules have been reported. ^{129}Xe analysis has been applied to the study of clathrates (*L159, L160*).

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SOLID BIOLOGICAL SYSTEMS

PAR150
SEN03 1 SEN06 7 SEN09 17 4 SEN12 14 8 SEN15 17 4 14 SEN18 20 5 SEN21 14 4 12 20 SEN24 28 8 SEN27 16 6 SEN30 14 5 SEN33 12 SEN36 7 3

Solid Biomolecules. The resolution available in NMR studies of solid proteins and nucleic acids cannot hope to approach those of analogous studies in solutions. One reason for this is the lack of conformational averaging in typical powder samples. Bryant has shown that the resolution obtained in ^{13}C CP/MAS spectra of lyophilized lysozyme can be improved by partial hydration (*M1*). Solid-state studies of biomolecules are motivated by reasons similar to those that motivate other such investigations, low solubility, conformational change upon dissolution, etc. The resolution problem is frequently dealt with by labeling, or sometimes double labeling, the structural feature of interest. The development of MAS experiments that preserve dipolar couplings has afforded the opportunity for doing distance determinations that cannot conveniently be done crystallographically for a lack of suitable crystals. One such method is rotational resonance, which is applicable for measuring internuclear distances from homonuclear dipolar couplings. Griffin and co-workers have used rotational resonance to identify the configuration of retinal in bacteriorhodopsin (*M2*). Another very promising high-resolution technique for distance determinations is the REDOR experiment. Schaefer and co-workers technique for distance determinations is the REDOR experiment. Schaefer and co-workers have used that technique to detect cross-links

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PAR150
in insect cuticle (*M3*).

PAR153
Solid-state NMR spectroscopy has also been applied to study dynamics in biomolecules including DNA duplex oligomers (*M4, M5*), a DNA intercalation complex (*M6*), proteins and peptides (*M7–M9*), and model cell membranes (*M10*). Principal components of chemical shift tensors in biomolecules have been measured in an effort to obtain more structural information than that afforded by isotropic shifts alone (*M11, M12*).

PAR156
Other work in solid-state NMR spectroscopy of biomolecules has been covered in reviews on DNA (*M13*) and proteins (*M14*).

TXT45
SOLID POLYMERS

PAR159
Solid state NMR techniques such as MAS, wide-line ^2H, and relaxation studies are widely applied to characterize the morphology and dynamics of polymers. The structure of the polymer is typically known, but this can be the main issue in studies of resins and cross-linking.

PAR162
Studies of local motions in polymers have been reviewed (*N1*). Harris has observed nuclear Overhauser enhancements in polymer films (*N2*). Tonelli has studied polymer chains confined to channels in clathrates (*N3, N4*). Spiess has advanced the use of 2-D NMR spectroscopy to study ultraslow chain motion (*N5*).

PAR165
Selected applications of NMR spectroscopy to solid polymers are probably most usefully cited on the basis of class of material. Several studies by Maciel looked at the chemistry of resins (*N6–N9*). Diverse polymers of biological origin have been studied (*N10–N14*). Synthetic polymer classes examined by NMR spectroscopy included polycarbonates (*N15–N17*), polystyrenes (*N18–N22*), Nylons (*N23, N24*), PEEK (*N25*), liquid crystalline polymers (*N26, N27*), and polyethylene (*N28–N30*). Reports were published on the study of conductive polymers (*N31–N33*) and polyacetylenes (*N34, N35*). Urethanes have been investigated (*N36, N37*) as have composites (*N38, N39*) and a number of blends (*N40–N47*). The effects of small molecules in polymers have been characterized (*N48–N50*), and the ubiquitous technique of ^{129}Xe NMR spectroscopy has been applied to polymers (*N51, N52*).

TXT48
SOLID INORGANIC COMPOUNDS

PAR168
The application of solid state NMR spectroscopy to problems in inorganic chemistry continues to expand. The diversity of elements and problems of interest in that field would seem to present a challenge to the organization of the relevant NMR literature. Much of it, however, can be grouped into one of several categories. There were a number of studies which related solid-state NMR and X-ray crystallographic data (*O1–O9*). Studies of stereochemical nonrigidity in solid inorganic compounds have become almost as common as analogous studies in solutions (*O10–O16*). A number of studies have been directed at measuring chemical shift tensors and/or indirect spin–spin coupling tensors in inorganic compounds (*O17–O23*). Variable-temperature studies have probed magnetic properties (*O24, O25*), and an improvement in the resolution of spectra of paramagnetic lanthanide complexes has been reported (*O26*). Phosphorus multiple bonds have been characterized (*O27, O28*). NMR analysis continues to be applied to the study of phase transitions of various sorts (*O29–O31*).

PAR171
Several other studies of interest did not fit into any easily contrived categories. The ^{127}I MAS spectrum of βAgI is very sensitive to Ag^+ diffusion over a wide temperature range (*O32*). A single-crystal ^{23}Na study of the sodide Na^+(cryptand [2.2.2])·Na^- has been reported (*O33*). ^{199}Hg MAS NMR spectroscopy has been used to probe coordination number in Hg(II) complexes (*O34*), and ^{27}Al MAS has characterized $AlCl_3$–THF complexes (*O35*).

TXT51
INORGANIC MATERIALS

PAR174
Some of the more creative applications of NMR spectroscopy to inorganic materials included Eckert's studies of non-oxide glasses and related materials (*P1–P10*), Stebbins' studies of silicates at extremes of temperature and pressure (*P11–P13*), and the application of DAS and DOR to ^{17}O analysis of solid silicates (*P14*). MAS NMR analysis was also applied to silicates and aluminates (*P15–P20*). Other classes of materials investigated included semiconductors (*P21–P23*), ceramics (*P24–P28*), and inorganic polymers (*P29–P33*). Clays and other layered materials were also studied (*P34–P43*). High-resolution ^{27}Al spectra of small particles of AF_3 were

TXT51
PAR174

obtained with line narrowing by Brownian motion in a liquid phase (*P44*).

TXT54

FULLERENES

PAR177

The discovery and isolation of C_{60}, C_{70}, and a growing number of other fullerenes is probably the most interesting development in chemistry in recent memory, certainly since the discovery of high-T_c superconductors (see below). Early success in fullerene research was controlled to a large extent by access to materials, so it is not surprising that the first NMR papers on C_{60} came from groups at the IBM Almaden Research Center (*Q1*) and AT&T Bell Laboratories (*Q2*). Solid C_{60} undergoes rapid isotropic motion at room temperature that averages the ^{13}C chemical shift anisotropy (*Q1*, *Q2*). This motion is quenched at 77 K, and it was possible to measure principal components of the ^{13}C chemical shift tensor of 220, 186, and 40 ppm (*Q1*). These values compare favorably to a theoretical calculation (*Q3*). As mentioned in the Introduction and Scope, Yannoni and co-workers used the Carr-Purcell sequence to measure bond lengths in ^{13}C-enriched C_{60} at 77 K (*Q4*). These values were 1.45 ± 0.015 and 1.40 ± 0.015 Å.

PAR180

Fullerenes have proven to be far more reactive than was originally believed, and a systematic chemistry is beginning to emerge. NMR studies of osmylated C_{60} (*Q5*) and polymethylated C_{60} (*Q6*) have recently been reported. ESR studies of C_{60}^- trapped in a zeolite (*Q7*) and the radical anion of C_{60} (*Q8*) have also appeared.

TXT57

HIGH-T_C SUPERCONDUCTORS

PAR183

NMR experiments are contributing to the understanding of magnetic properties of high transition temperature ceramic superconductors. Studies of the $YBa_2Cu_3O_{6+x}$ series were reviewed by Walstedt and Warren (*R1*). A number of superconductor materials have been investigated by ^{63}Cu and/or ^{65}Cu (*R2–R7*), ^{17}O (*R8–R11*), ^{205}Tl (*R12, R13*), or ^{89}Y (*R14–R16*) spectroscopy. Relationships between theories of superconductivity and NMR experiments have also been explored (*R17–R19*).

TXT60

GAS-PHASE NMR SPECTROSCOPY

PAR186

NMR studies of gases are generally geared toward understanding the energetics of collisions. Gas-phase NMR studies published during the period covered by this review included studies related to conformational processes (*S1*, *S2*), nuclear shielding (*S3–S5*), and relaxation phenomena (*S6–S8*).

TXT63

IMAGING, MICROSCOPY, AND DIFFUSION

PAR189

NMR imaging has had a profound effect on clinical medicine, and it is having an effect on chemical problems involving spatial inhomogeneities. One of the most intriguing examples of this to catch my eye was the detection of chemical waves in the Mn^{2+}-catalyzed Belousov-Zhabotinsky reaction (*T1*). Clinical aspects of imaging and spatially-localized spectroscopy are outside the scope of this review, but the interested reader is referred to a good introductory article (*T2*). Some of the most common applications of NMR imaging in chemistry and engineering include diffusion of solvents in swollen polymers (*T3–T5*) and oil and/or water in porous rock (*T6–T8*).

PAR192

In the limit of smaller sample sizes and high spatial resolution, NMR imaging is frequently referred to as NMR microscopy (*T9–T11*). Since microscopy can be performed in standard wide-bore (89-mm) magnets, it is likely to grow in popularity among chemists. Volume-localized spectroscopy is important for slice selection in in vivo spectroscopy or other problems requiring selective excitation of a spatial region near the coil (*T12–T14*). This can be possible even for solids (*T15–T17*). Solid-state NMR imaging has been reviewed (*T18*), and several improvements or applications of the technique have been reported (*T19–T26*). In spite of the apparent difficulty of imaging a rotating solid, the advantages of line narrowing have motivated study (*T27–T29*). NQR imaging has also been reported (*T30*).

PAR195

Field gradients are also used in NMR studies of diffusion, dating from the work of Stejskal and Tanner in the mid-60s. The availability of pulsed-field gradient coils has extended the range of this method. Several applications of diffusion methods were reported (*T31–T36*) and others were cited in the section on Adsorption Phenomena and Catalysis. The role of diffusion in imaging studies should not be neglected (*T37*, *T38*).

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PAR198

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